FORM PTO-1390 (Modified) (REV 10-95) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER	U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR)
JMYT-V00200	To be assigned 09/380864

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/GB98/00705	9 MAR 1998 (09.03.98)	10 MAR 1997 (10.03.97)

TITLE OF INVENTION

EMISSION CONTROL SYSTEM FOR A LEAN-BURN INTERNAL COMBUSTION ENGINE

APPLICANT(S) FOR DO/EO/US

TWIGG, Martyn Vincent

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
   a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ A copy of the International Search Report (PCT/ISA/210).
8. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☒ have not been made and will not be made.
9. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
10. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)). (UNEXECUTED)
11. ☒ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

Items 13 to 18 below concern document(s) or information included:

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
    A **SECOND** or **SUBSEQUENT** preliminary amendment.
16. ☐ A substitute specification.
17. ☐ A change of power of attorney and/or address letter.
18. ☒ Certificate of Mailing by Express Mail
19. ☐ Other items or information:

514 Rec'd PCT/PTO 09 SEP 1999

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 1.5)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
09/380864 To be assigned	PCT/GB98/00705	JMYT-V00200

20. The following fees are submitted:.

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :

		CALCULATIONS PTO USE ONLY
☒ Search Report has been prepared by the EPO or JPO	$840.00	
☐ International preliminary examination fee paid to USPTO (37 CFR 1.482)	$670.00	
☐ No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2))	$760.00	
☐ Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2) paid to USPTO	$970.00	
☐ International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4)	$96.00	

ENTER APPROPRIATE BASIC FEE AMOUNT =	$840.00	
Surcharge of **$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).	$0.00	

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	12 - 20 =	0	x $18.00	$0.00	
Independent claims	2 - 3 =	0	x $78.00	$0.00	
Multiple Dependent Claims **(check if applicable)**.			☒	$260.00	
TOTAL OF ABOVE CALCULATIONS =				$1,100.00	
Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) **(check if applicable).**			☐	$0.00	
SUBTOTAL =				$1,100.00	
Processing fee of **$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (f)). +				$0.00	
TOTAL NATIONAL FEE =				$1,100.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) **(check if applicable).**			☐	$0.00	
TOTAL FEES ENCLOSED =				$1,100.00	
				Amount to be: refunded	$
				charged	$

☒ A check in the amount of **$1,100.00** to cover the above fees is enclosed.

☐ Please charge my Deposit Account No. in the amount of to cover the above fees.
A duplicate copy of this sheet is enclosed.

☒ The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment to Deposit Account No. **18-0350** A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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Paul F. Prestia
SIGNATURE

Paul F. Prestia
NAME

23,031
REGISTRATION NUMBER

September 9, 1999
DATE

JMYT-V00200

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	Martyn Vincent Twigg	: Art Unit:
Serial No.:	To be Assigned	: Examiner:
Filed: :	Herewith	:
FOR: :	EMISSION CONTROL SYSTEM	:
	FOR A LEAN-BURN INTER-	:
	NAL COMBUSTION ENGINE	:

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231
Box PCT

S I R :

Prior to examination, pleased amend the above-identified application as follows.

IN THE SPECIFICATION:

On page 1, after the title, please insert the following sentence:

-- This application is the U.S. national-phase application of PCT International Application No. PCT/GB98/00705.--

IN THE CLAIMS:

Please amend the following claims:

4. (Amended) A system according to [any of claims]claim 1 [to 3]or 2, wherein both the first and second catalyst systems comprise platinum.

8. (Amended) A process according to claim 5[,]or 6 [or 7], wherein the engine is in a vehicle.

REMARKS

The Assistant Commissioner is hereby authorized to charge payment to Deposit Account No. 18-0350 of any fees associated with this communication.

Respectfully submitted,

Paul F. Prestia, Reg. No. 23,031
Attorney for Applicant

Dated: September 9, 1999

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EXPRESS MAIL Mailing Label Number: **EL 442 467 523 US**
Date of Deposit: **September 9, 1999**

I hereby certify that this paper and fee are being deposited, under 37 C.F.R. § 1.10 and with sufficient postage, using the "Express Mail Post Office to Addressee" service of the United States Postal Service on the date indicated above and that the deposit is addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231.

Kathleen Libby

EMISSION CONTROL SYSTEM FOR A LEAN-BURN INTERNAL COMBUSTION ENGINE

This invention concerns improvements in emissions control systems, and more especially it concerns improvements in emissions control for engines operating at lean air/fuel ratios, *ie* air/fuel ratios greater than 14.7, generally in the range 19-50.

It will be appreciated that with lean-burn engines of various types, including particularly diesel, lean-burn gasoline and direct injection gasoline engines, the control of NOx tends to be difficult. This is understandable in that the exhaust gases contain relatively high amounts of oxygen and hence the removal of NOx involves reduction of NOx to N_2 in an overall oxidising atmosphere. Prior proposals have involved storage of NOx in the emission control system until a time when the exhaust gas contains relatively less oxygen, that is until the engine is running "rich", *eg* during acceleration. Another proposal is to store unburnt hydrocarbon until a point at which it can be released to contribute to NOx reduction. There remains the need, however, for yet further systems and strategies to achieve control of NOx emissions under lean conditions.

The present invention provides a novel emission control system for a lean-burn internal combustion engine, comprising a first catalyst system comprising platinum group metal and having relatively high selectivity for NOx reduction, and a second catalyst system having high activity for the oxidation of hydrocarbons and carbon monoxide. By platinum group metal is meant platinum and/or palladium and/or rhodium.

The invention also provides a process for the control of emissions from a lean-burn internal combustion engine, comprising passing the exhaust from the engine over a first catalyst system comprising platinum group metal and having relatively high selectivity for NOx reduction, and then passing the product gases exiting from said first catalyst system over a second catalyst system having high activity for the oxidation of hydrocarbons and carbon monoxide.

By selectivity for NOx reduction is meant the ratio of %NOx conversion to % hydrocarbon conversion. The catalyst system having relatively high such selectivity has a

selectivity of at least 0.2, preferably at least 0.3, especially at least 0.4; this is as measured at a temperature of 230°C, a space velocity of 25000hr^{-1} and a hydrocarbon:NOx input ratio of 3:1 counting the hydrocarbon as equivalent propane. The catalyst system having high activity for the oxidation of hydrocarbons and carbon monoxide has, as measured under the same conditions, a % hydrocarbon conversion of greater than 80%, preferably greater than 90%; it has, as measured under the same conditions, a % carbon monoxide conversion of greater than 70%, preferably greater than 80%, especially greater than 90%, particularly greater than 95%. Defining the catalyst systems according to measurement under these conditions does not mean of course that they are necessarily operated under these conditions. In a particular embodiment, the first catalyst system is such that the exhaust gases from the engine flow over it at a low space velocity, particularly below 40000hr^{-1}. The second catalyst system is usually such that the exhaust gases from the engine flow over it at a space velocity of 40000-80000hr^{-1}. The first catalyst system usually contains platinum. The second catalyst system usually contains platinum. Thus, in a particular embodiment each contains platinum. For use, the first catalyst system can be mounted ahead of the second catalyst system in the exhaust apparatus of the engine. The present engine is preferably in a vehicle, for example a passenger car or heavy duty truck.

The skilled person may apply the present invention in a variety of ways. The first catalyst system may be, for example, a relatively low loading of catalytically active component on a substrate, optionally in combination with components that can retain NOx and/or reducing species, such as zeolite or like absorbents, or alkaline earth metal compounds. We have discovered that reducing the loading of catalytically active component (comprising platinum group metal, particularly platinum, optionally in the presence of base metal components) compared to conventional exhaust gas catalysts, serves to increase the selectivity of the catalyst system towards NOx reduction. The first catalyst system can contain for instance platinum group metal, particularly platinum, in amount less than 30g/ft^3 (30g per 0.028m^3).

In a particular embodiment, the first catalyst system provides a low space velocity. Normal space velocities for exhaust catalysts systems are 40000-80000hr^{-1}. A lower space

velocity may be achieved readily in practice by increasing the volume of the catalyst, or by utilising two catalyst "bricks" in parallel.

The second catalyst system is required to be of high oxidation activity. Such catalyst need not have NOx selectivity, but must be capable of oxidising hydrocarbons and carbon monoxide under the reaction conditions, to the desired extent, usually so as to satisfy emission standards regulations. Normal space velocities may be used. The second catalyst system usually comprises platinum group metal, particularly platinum, optionally in the presence of base metal components. A suitable oxidation catalyst comprises platinum on a high surface area support, optionally with other components which promote such oxidations.

The skilled person is very familiar with conventional exhaust gas catalyst technology. Generally, a support which is a honeycomb-type extruded ceramic or wound metal monolith or "brick" is coated with a surface area-enlarging washcoat, for example a washcoat consisting of or comprising alumina. Deposited onto the washcoat is a coating of one or more catalytic components, optionally with one or more other components such as ceria, zirconia, zeolite or the like, and the catalyst may be multi-component deposited in discrete layers or some components may be layered, with other components distributed throughout such layers. In the present invention, the actual catalysts chosen, and their construction, are not critical providing they meet the criteria stated.

It is now well established that carbon monoxide and hydrocarbons play a part in the reduction of NOx. For example, taking the hydrocarbon as C_3H_6, the following reactions could take place:

$$18NO + 2C_3H_6 \rightarrow 9N_2 + 6CO_2 + 6H_2O$$

$$2NO + 2CO \rightarrow N_2 + 2CO_2$$

The molar ratios required for NOx reduction, namely C_3H_6 to NO and CO to NO, are exceeded over the European test cycle on average with a diesel engine. However, there

is competition between NOx and oxygen for the reducing species, and normally only quite low NOx conversions, (*eg* much less than 10%) are achieved in the European test cycle.

It may be preferred to increase NOx conversion under certain conditions by increasing the relative quantity of hydrocarbons in the exhaust. For example, injection of fuel into the exhaust upstream of the first catalyst system may be used. Alternatively, hydrocarbon storage using zeolites or the like, may be useful. It will be appreciated that there would be a small fuel consumption penalty if fuel injection into the exhaust is used. The high activity second catalyst system is readily capable of catalysing the oxidation of any excess hydrocarbons under the lean conditions.

Conventional catalyst manufacturing technology may be used.

The first and second catalyst systems may be mounted in a single "can" in the exhaust system, or they may be separated by a length of exhaust pipe.

The present engine is generally a diesel, lean-burn gasoline or direct injection gasoline engine.

The present invention is illustrated by the following Tests.

Test 1

The increase of NOx selectivity corresponding to decreasing platinum loading was shown for a standard 6in (15.2cm) catalyst brick. Exhaust from a 1.9 litre turbo direct injection diesel bench engine, operating at steady state conditions was used. NOx selectivity is measured as % NOx conversion at 230°C/% hydrocarbon conversion at 230°C. The results are shown in Table 1 below.

TABLE 1

Pt loading (g/ft^3) ($g/0.028m^3$)	NOx selectivity at 230°C
10	1.00
25	0.40
50	0.34
75	0.33
100	0.31

A reduced loading of catalyst therefore improves selectivity.

Test 2

The increase in NOx conversion, at a constant platinum loading (1.5g) *per* catalyst brick, by decreasing space velocity and reducing loading in g/unit volume was measured. The same engine and conditions was used as in Test 1. The NOx conversion was measured with "raw" exhaust from the engine ("Passive") and with the addition of hydrocarbon (HC) into the exhaust to yield a HC3:NOx ratio of 2.0:1. HC3 means that the hydrocarbon is counted as equivalent propane.

TABLE 2

Catalyst length inches (cm)	Maximum NOx Conversion (%)	
	Passive	Added HC
1 (2.5)	6	13
2 (5.1)	12	17
3 (7.6)	16	22
4 (10.1)	18	25
5 (12.7)	22	29
6 (15.2)	25	33

It can clearly be seen that increasing catalyst length and hence decreasing space velocity is beneficial in overall NOx conversion.

EXAMPLE 1

A 1996 model passenger car with a 2.5 litre turbo direct injection diesel engine was used with several different exhaust catalyst systems, for standard EUDC emission tests (Extra Urban Driving Cycle emission tests of the European Union). The results are shown in Table 3 below.

TABLE 3

Catalyst System

	HC	CO	NOx	HC+ NOx	PM (4)	%NOx
No Catalyst	0.322	1.034	0.394	0.715	0.089	0.0
OEM Catalyst (1)	0.196	0.913	0.389	0.585	0.081	1.3
New Catalyst (2)	0.067	0.336	0.332	0.399	0.079	15.7
Lean-NOx Catalyst (3)	0.054	0.495	0.294	0.347	0.073	25.4
Lean-NOx Catalyst + Oxidation Catalyst (5)	0.037	0.237	0.292	0.329	0.077	25.9

Notes:

(1) OEM (Original Equipment Manufacturer) catalyst, 6in (15.2cm) long, with 46g/ft^{-3} (g per 0.028m^3) Pt.

(2) Advanced oxidation catalyst, 6 in (15.2cm) long with 40g/ft^{-3} (g per 0.028m^3) Pt.

(3) Lean-NOx catalyst, 12 in (30.5cm) long, with 25g/ft^{-3} (g per 0.028m^3) Pt.

(4) PM = Particulate Matter, g/km

(5) Lean-NOx catalyst, 9in (22.9cm) long, with 25g/ft^{-3} (g per 0.028m^3) Pt, followed by oxidation catalyst, 3in (7.6cm) long, with 100g/ft^{-3} (g per 0.028m^3) Pt.

All the catalysts used were fresh, *ie* without ageing.

It can readily be seen that the low loading, low space velocity Lean-NOx Catalyst is very effective in converting NOx, and that the combination according to the invention is remarkably effective.

SUBSTITUTE SET OF CLAIMS

1. An emission control system suitable for a lean-burn internal combustion engine, comprising a first up-stream catalyst system comprising a platinum group metal and a second downstream catalyst system comprising a platinum group metal characterised in that said first and second catalyst systems do not include silver or tungsten, further characterised in that said first catalyst systems comprises platinum and/or palladium and/or rhodium and has a ratio of $\%NO_x$ conversion to % hydrocarbon conversion of at least 0.2 as measured at a temperature of 230°C a space velocity of 25,000 hr^{-1} and a hydrocarbon:NO_x input ratio of 3:1 counting the hydrocarbon as equivalent propane, and further characterised in that said second catalyst system has as measured under the same conditions, a % hydrocarbon conversion greater than 80% and a % carbon monoxide conversion greater than 70%.

2. A system according to claim 1, so designed and constructed that when connected to an engine, the exhaust gas flow over the first catalyst system is at a space velocity of below 40,000hr^{-1}.

3. A system according to claim 1 or 2, so designed and constructed that when connected to an engine, the exhaust gas flow over the second catalyst system is at a space velocity of from 40,000 to 80,000hr^{-1}.

4. A system according to any of claims 1 to 3, wherein both the first and second catalyst systems comprise platinum.

5. A process for the control of emissions from the exhaust gases from a lean-burn internal combustion engine by passing said exhaust gases over a first upstream catalyst system and a second downstream catalyst system, characterised in that said first and second catalyst systems do not include silver or tungsten, further characterised in that said first catalyst systems comprises platinum and/or palladium and/or rhodium and has a ratio of $\%NO_x$ conversion to % hydrocarbon conversion of at least 0.2 as measured at a temperature of 230°C a space velocity of 25,000 hr^{-1} and a hydrocarbon:NO_x input ratio of 3:1 counting the hydrocarbon as equivalent propane, and further characterised in that said second catalyst system has, as measured under the

same conditions, a % hydrocarbon conversion greater than 80% and a % carbon monoxide conversion greater than 70%.

6. A process according to claim 5, characterised in that the exhaust gases are passed over the first catalyst system at a space velocity below 40,000 hr^{-1}.

7. A process according to claim 5 or 6, characterised in that the exhaust gases are passed over the second catalyst system at a space velocity of from 40,000 to 80,000 hr^{-1}.

8. A process according to claim 5, 6 or 7, wherein the engine is in a vehicle.

Declaration and Power of Attorney For Patent Application
English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled
EMISSION CONTROL SYSTEM FOR A LEAN-BURN INTERNAL COMBUSTION ENGINE ,
the specification of which is attached hereto unless the following box is checked:

☒ was filed on March 9, 1998 as
United States Application Number or PCT International Application Number PCT/GB98/00705
and was amended on February 27, 1999 and September 9, 1999 (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)			Priority Not Claimed
9705010.8	GB	March 10, 1997	☐
(Number)	(Country)	(Day/Month/Year Filed)	
—	—	—	☐
(Number)	(Country)	(Day/Month/Year Filed)	

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below.

—	—
(Application Number)	(Filing Date)
—	—
(Application Number)	(Filing Date)

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s), or 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

___ (Application Number)	___ (Filing Date)	___ (Status - patented, pending, abandoned)
___ (Application Number)	___ (Filing Date)	___ (Status - patented, pending, abandoned)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

Paul F. Prestia	Reg. No. 23,031	Lawrence E. Ashery	Reg. No. 34,515	Mark J. Marcelli	Reg. No. 36,593
Allan Ratner	Reg. No. 19,717	Christopher R. Lewis	Reg. No. 36,201	Joshua L. Cohen	Reg. No. 38,040
Andrew L. Ney	Reg. No. 20,300	Robert L. Andersen	Reg. No. 25,771	Christopher J. Dervishian	Reg. No. 42,480
Kenneth N. Nigon	Reg. No. 31,549	Daniel N. Calder	Reg. No. 27,424	Jack J. Jankovitz	Reg. No. 42,690
Kevin R. Casey	Reg. No. 32,117	Louis W. Beardell, Jr.	Reg. No. 40,506		
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor (given name, family name) **Martyn Vincent TWIGG**

Inventor's signature Martyn Twigg. Date 17 November 99

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Full name of second joint inventor, if any (given name, family name) ___

Second Inventor's signature _____ Date _____

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☐ Additional inventors are being named on separately numbered sheets attached hereto.